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Polymeric Coordination Compounds Derived from Transition Metal(II) with Tetradentate Schiff-base: Synthetic, Spectroscopic, Magnetic and Thermal Approach

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The polymeric coordination compounds have been synthesized using the dipotassium salt of N,N'-di(carboxymethylene)terephthalaldehydediimine (K_2SB^1) or N,N'-di(carboxyethylene)terephthalaldehydediimine (K_2SB^2) with manganese(II), cobalt(II), nickel(II), copper(II), zinc(II), and cadmium(II). The ligands have been characterized by 1H -NMR, and ^{13}C -NMR spectra. The polychelates have been characterized by elemental analyses, magnetic measurements, thermogravimetric analyses, electronic spectra and infrared spectra. The coordination compounds are colored, and the central metal ions are octahedrally coordinated with two water molecules and the Schiff bases. The Schiff bases act as di-negative tetradentate ligands, in which bonding occurs through two oxygen and two nitrogen atoms. The polymers are insoluble in all common organic solvents such as acetone, methanol, ethanol, N,N-dimethylformamide and dimethylsulfoxide.

Keywords: coordination polymers; transition metal ions; Schiff bases; spectroscopic; thermal and magnetic studies

1 Introduction

Schiff bases are used as biological models and are known to possess tuberculostatic (1), fungicidal, bacteriostatic (2), anti-tumor and antiviral activities. Schiff bases containing polyfunctional groups have produced stable metal complexes of transition, lanthanide and actinide metal ions (3). Schiff base complexes have played a significant role in analytical chemistry, biochemistry and agricultural chemistry. In addition to the above discussion, the coordination polymers, derived from the Schiff bases are also the center of interest, are usually known for their thermal stability (4, 5). They are defined as materials in which metal ions are linked together with di- or polyfunctional ligands. Much attention has been paid to porous high-dimensional coordination polymers, because of their potential zeolite like applications (6). However, a number of equally important applications of coordination polymers have been reported such as their use as non-linear optical devices, catalysts, solar energy converters, and their ability to remove SO_x and NO_x from the environment (7–10). The synthesis and characterization of

coordination polymers have been the subject of active research in our laboratory over the past few years (11–13). In this communication, we report the synthetic, spectroscopic, magnetic and thermal properties of the coordination polymers of Mn(II), Co(II), Ni(II), Cu(II), Zn(II), and Cd(II) with tetradentate ligands. The structures of the ligands are shown in Figure 1.

2 Experimental

2.1 Materials and Instruments

All the chemicals used were of analytical grade. Terephthalaldehyde, glycine, L-alanine, potassium hydroxide and metal acetates were purchased from E. Merck Ltd., Mumbai, India. The organic solvents were purified by standard methods (14). The infrared spectra were recorded on a FT-IR Nicolet 400D spectrophotometer as KBr pellets. Carbon, hydrogen, and nitrogen were analyzed with a model 240 Perkin-Elmer elemental analyzer. The metal content of the coordination polymers were determined by EDTA titration (15) after decomposing the organic matter with a mixture of perchloric, sulfuric and nitric acids [1:1.5:2.5]. Thermogravimetric analyses were obtained with a model 5000/2960 SDTA, TA Instruments, USA. The reflectance spectra of

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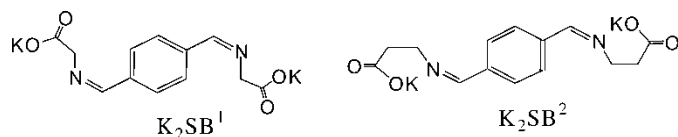


Fig. 1. Structure of the ligands.

the coordination polymers were recorded in the range, 1700–350 nm as MgO discs on a Beckman DK-2A spectrophotometer. The magnetic moments were obtained by the Gouy's method using mercury tetrathiocyanatocobaltate(II) as a calibrant ($\chi_g = 16.44 \times 10^{-6}$ c.g.s. units at 20°C). Diamagnetic corrections were made using Pascal's constant (16).

2.2 Synthesis of the Ligands

A solution of potassium hydroxide (20 mmol, 1.12 g) in 50 ml absolute alcohol was added to glycine (20 mmol, 1.5 g) or L-alanine (20 mmol, 1.78 g). The mixture was stirred until all the solids dissolved in the ethanol-KOH solution. Then, an ethanolic solution (100 ml) of terephthalaldehyde (10 mmol, 1.34 g) was added quickly. The mixture was heated on a water bath for 1 h. A yellow crystalline solid was obtained on cooling to room temperature. The product was filtered, washed with ethanol, and dried in a vacuum desiccator.

2.3 Synthesis of the Coordination Polymers

The methanolic solution of metal acetate (10 mmol, diluted to 100 ml) was added slowly to a methanolic solution of K_2SB^1 (10 mmol, 3.24 g diluted to 100 ml) or K_2SB^2 (10 mmol, 3.52 g diluted to 100 ml) in 1:1 mole ratio. The mixture was heated for 8 h at 80°C to ensure complete precipitation. The solution was then allowed to cool overnight at room temperature. The coordination polymers were filtered, washed with methanol and dried in air. All the coordination polymers are insoluble in all common organic solvents like acetone, ethanol, chloroform, methanol, benzene, DMF, and DMSO.

3 Results and Discussions

Elemental analyses, colors, yields, melting points, and magnetic moments are summarized in Table 1. The elemental analyses of the Schiff bases K_2SB^1 and K_2SB^2 and their coordination polymers are in agreement with the proposed formulae. All of the coordination polymers exhibit 1:1 metal-to-ligand stoichiometry. The polymeric compounds were insoluble in common organic solvents and, therefore, precluded the determination of molecular weights. Elemental analyses, physical properties and infrared data provide good evidence that the chelates are polymeric in nature (11, 17). The suggested structures of the coordination polymers are shown in Figure 2.

Table 1. Analytical data of the coordination polymers^a

Empirical formula of monomer unit	Formula weight of monomer unit	Color	% Found (Calcd.)				M.p. (°C)	μ_{eff} (B.M.) calcd.	% Yield
			C	H	N	Metal			
K_2SB^1 $C_{12}H_{10}K_2N_2O_4$	324.31	Yellow	44.42 (44.44)	3.09 (3.08)	8.61 (8.63)	—	141	—	60
K_2SB^2 $C_{14}H_{14}K_2N_2O_4$	352.33	Yellow	47.70 (47.72)	3.94 (3.97)	7.92 (7.94)	—	> 360	—	63
$[Mn(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}MnN_2O_6$	337.06	Brown	42.72 (42.75)	4.17 (4.15)	8.31 (8.30)	16.21 (16.29)	> 360	5.67 (5.92)	38
$[Co(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}CoN_2O_6$	341.05	Pink	42.20 (42.26)	4.13 (4.10)	8.25 (8.20)	17.16 (17.27)	> 360	3.64 (3.87)	42
$[Ni(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}NiN_2O_6$	340.81	Green	42.24 (42.28)	4.17 (4.10)	8.26 (8.21)	17.16 (17.22)	> 360	2.97 (2.83)	45
$[Cu(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}CuN_2O_6$	345.66	Green	41.71 (41.69)	4.09 (4.05)	8.13 (8.10)	18.29 (18.38)	> 360	1.93 (1.73)	41
$[Zn(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}ZnN_2O_6$	347.51	Yellow	41.40 (41.47)	4.09 (4.03)	8.03 (8.05)	18.70 (18.81)	> 360	—	44
$[Cd(SB^1)(H_2O)_2]_n$ $C_{12}H_{14}CdN_2O_6$	394.53	Yellow	36.57 (36.53)	3.52 (3.55)	7.03 (7.09)	28.47 (28.49)	> 360	—	40
$[Mn(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}MnN_2O_6$	365.08	Brown	46.09 (46.05)	4.96 (4.93)	7.62 (7.67)	15.09 (15.04)	> 360	6.08 (5.92)	43
$[Co(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}CoN_2O_6$	369.07	Brown	45.52 (45.55)	4.82 (4.87)	7.51 (7.58)	15.90 (15.96)	> 360	4.03 (3.87)	39
$[Ni(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}NiN_2O_6$	368.83	Green	45.53 (45.58)	4.86 (4.88)	7.55 (7.59)	15.96 (15.91)	> 360	2.90 (2.83)	42
$[Cu(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}CuN_2O_6$	373.68	Green	44.92 (44.99)	4.82 (4.81)	7.45 (7.49)	16.98 (17.00)	> 360	1.80 (1.73)	46
$[Zn(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}ZnN_2O_6$	375.53	Yellow	44.72 (44.77)	4.76 (4.79)	7.44 (7.45)	17.38 (17.41)	> 360	—	40
$[Cd(SB^2)(H_2O)_2]_n$ $C_{14}H_{18}CdN_2O_6$	422.55	Yellow	39.77 (39.79)	4.23 (4.26)	6.60 (6.62)	26.67 (26.60)	> 360	—	43

^a K_2SB^1 = Dipotassium salt of N,N'-di(carboxymethylene)terephthalaldehydediimine, K_2SB^2 = Dipotassium salt of N,N'-di(carboxyethylene)terephthalaldehydediimine.

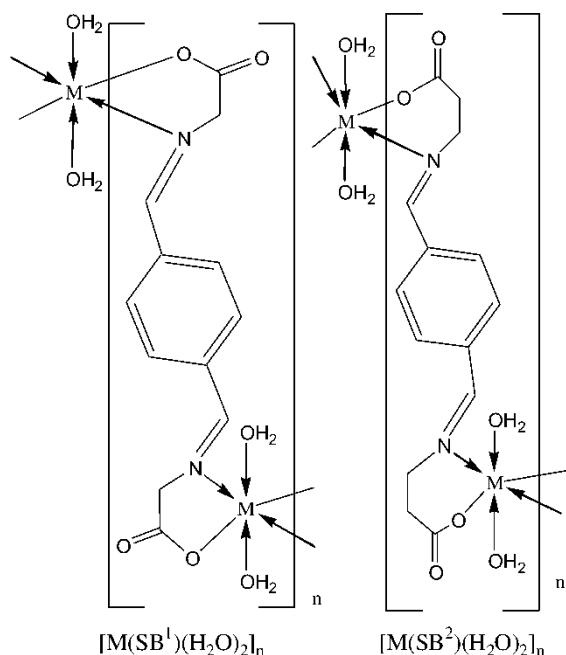


Fig. 2. Proposed structure of the coordination polymers.

3.1 $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ Spectra

The $^1\text{H-NMR}$ spectra and $^{13}\text{C-NMR}$ spectra of the ligands have been taken in DMSO-d_6 . The $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectral data are reported along with the possible assignment in Table 2. The $^1\text{H-NMR}$ spectra of ligands exhibit peaks at 7.7–7.8 ppm assigned to the aromatic protons. The singlet peak appeared at 8.2 and 8.3 ppm are assigned to azomethine protons ($-\text{CH}=\text{N}-$). The peaks appeared at 3.2–4.1 ppm is assigned to the aliphatic protons. In the case of $^{13}\text{C-NMR}$ spectra, peaks observed at 125.2–139.6 ppm are assigned to aromatic carbons, peaks at 165.8 and 167.4 ppm assigned to $-\text{CH}=\text{N}-$ carbons, peak at 170.3 and 172.6 ppm assigned to $>\text{C}=\text{O}$ group carbons, while peaks at 34–53 ppm assigned to aliphatic carbons.

Table 2. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ data of ligands^a

Compounds	$^1\text{H-NMR}$	$^{13}\text{C-NMR}$
H_2SB^1	7.7–7.8(4H, Ar-H), 8.2(1H, CH=N), 3.9(4H, Aliphatic)	125.2–139.6(Aromatic), 53(C-N), 165.8(CH=N), 170.3(>C=O)
H_2SB^2	7.7–7.8(4H, Ar-H), 8.3(1H, CH=N), 3.2–4.1(8H, Aliphatic)	34(Aliphatic), 125.2–139.6(Aromatic), 45(C-N), 167.4(CH=N), 172.6(>C=O)

^a K_2SB^1 = Dipotassium salt of $\text{N,N}'$ -di(carboxymethylene)terephthalaldehydediimine; K_2SB^2 = Dipotassium salt of $\text{N,N}'$ -di(carboxyethylene)terephthalaldehydediimine.

3.2 IR Spectra

The IR spectra of the coordination polymers were carried out as KBr pellets. All the polymers exhibit a broad band in the 3400–3000 cm^{-1} region, which supports the presence of coordinated water molecules (18, 19). In addition, the rocking and wagging modes of $\nu(\text{O-H})$ are observed at 850 and 710 cm^{-1} , respectively (20–22). A comparison of the IR spectra of free ligands with those of their coordination polymers indicate a substantial shift in the $\nu(\text{C}=\text{N})$ stretching frequency. The decrease in the $\text{C}=\text{N}$ stretching frequencies has been attributed to the coordination of the metal ion via the azomethine nitrogen atom (23, 24). The presence of a sharp band in the region 525–535 cm^{-1} , for all the coordination polymers can be assigned to $\nu(\text{M-N})$ (25), which supports the involvement of nitrogen in coordination. The asymmetric and symmetric stretching frequencies of the carboxylate ions in the polymers are observed at 1595 cm^{-1} and 1400 cm^{-1} , respectively, and are shifted to lower frequencies (~ 1585 cm^{-1} and ~ 1390 cm^{-1}) (26–28). The medium intensity bands for $\nu(\text{M-O})$ (29) have been observed at 415–420 cm^{-1} , thereby confirming metal-to-oxygen coordination. Sharp bands at 750–780 and 1525–1535 cm^{-1} are due to aromatic $\nu(\text{C-H})$ (30) and $\nu(\text{C}=\text{C})$ (31), respectively, and bands in the region 1160–1170 cm^{-1} , are attributed to $\nu(\text{C-N})$.

3.3 Electronic Spectra

The electronic spectra of the coordination polymers have been studied in solid state. The electronic spectra provide information about the electronic structure. The electronic spectra of the Ni(II) coordination polymers gave three bands at $\sim 10,500$, $\sim 18,000$ and $\sim 24,000$ cm^{-1} , which have been ascribed to the $^3\text{A}_{2g}(\text{F}) \rightarrow ^3\text{T}_{2g}(\text{F})$ (ν_1), $^3\text{A}_{2g}(\text{F}) \rightarrow ^3\text{T}_{1g}(\text{F})$ (ν_2) and $^3\text{A}_{2g}(\text{F}) \rightarrow ^3\text{T}_{1g}(\text{P})$ (ν_3) transitions, respectively, and correspond to an octahedral geometry of the Ni(II) ion (32, 33). The electronic spectra of the Co(II) coordination polymers exhibit three bands at $\sim 9,200$ (ν_1), $\sim 17,900$ (ν_2) and $\sim 19,000$ (ν_3) cm^{-1} , which may reasonably be assigned (34) to the $^4\text{T}_{1g}(\text{F}) \rightarrow ^4\text{T}_{2g}(\text{F})$ (ν_1), $^4\text{T}_{1g}(\text{F}) \rightarrow ^4\text{A}_{2g}(\text{F})$ (ν_2), and $^4\text{T}_{1g}(\text{F}) \rightarrow ^4\text{T}_{1g}(\text{P})$ (ν_3) transitions and are consistent with an octahedral geometry for this metal ion. The electronic spectra of the Cu(II) coordination polymers exhibit one band at $\sim 15,500$ cm^{-1} assignable to the $^2\text{E}_g \rightarrow ^2\text{T}_{2g}$ transition, and correspond (35) to an octahedral geometry for Cu(II) ion. The electronic spectra of the Mn(II) coordination polymers (36) gave three weak absorption bands at $\sim 15,000$, $\sim 19,500$ and $\sim 24,500$ cm^{-1} . These bands have been assigned to the $^6\text{A}_{1g} \rightarrow ^4\text{T}_{1g}(^4\text{G})$ (ν_1), $^6\text{A}_{1g} \rightarrow ^4\text{T}_{2g}(^4\text{G})$ (ν_2) and $^6\text{A}_{1g} \rightarrow ^4\text{A}_{1g}, ^4\text{E}_g(^4\text{G})$ (ν_3) transitions, in order of increasing energy. The value of the ligand field splitting energy (10 Dq), Racah inter electronic repulsion parameter (B), nephelauxetic ratio (β), and ratio ν_2/ν_1 for the Co(II) and Ni(II) coordination polymers are summarized in Table 3.

Table 3. Electronic parameters of the Co(II) and Ni(II) coordination polymers^a

Coordination polymers	Observed bands (cm ⁻¹)				B	β	β°	10Dq
	ν_1	ν_2	ν_3	ν_2/ν_1				
[Co(SB ¹)(H ₂ O) ₂] _n	9,150	18,090	19,050	1.97	734	0.75	24	10,272
[Ni(SB ¹)(H ₂ O) ₂] _n	10,200	17,730	24,900	1.70	802	0.77	22	10,200
[Co(SB ²)(H ₂ O) ₂] _n	9,060	18,150	18,970	2.00	735	0.75	24	10,177
[Ni(SB ²)(H ₂ O) ₂] _n	10,370	17,525	25,020	1.69	762	0.74	26	10,370

^aK₂SB¹ = Dipotassium salt of N,N'-di(carboxymethylene)terephthalaldehydediimine. K₂SB² = Dipotassium salt of N,N'-di(carboxyethylene)terephthalaldehydediimine.

The ligand field splitting energy (10Dq), interelectronic repulsion parameter (B) and covalency factor (nephelauxetic ratio) (β) for the Co(II) and Ni(II) coordinating polymers have been calculated using the secular equations given by E.König [27].

For Co(II) Complexes

$$10Dq = 1/2 [(2\nu_1 - \nu_3) + \nu_3^2 + \nu_1 \nu_3 - \nu_1^2]^{1/2}$$

$$15B = \nu_3 - 2\nu_1 + 10Dq$$

$$\beta = B/B_0 \quad [B_0(\text{free ion}) = 971]$$

$$\beta^\circ = (1 - \beta) \times 100$$

For Ni(II) Complexes

$$10Dq = \nu_1$$

$$15B = (\nu_2 + \nu_3) - 3\nu_1$$

$$\beta = B/B_0 \quad [B_0(\text{free ion}) = 1030]$$

$$\beta^\circ = (1 - \beta) \times 100$$

3.4 Magnetic Measurements

The magnetic moment data of the coordination polymers are presented in the Table 1. The magnetic moments of the two Mn(II) coordination polymers, [Mn(SB¹)(H₂O)₂]_n and [Mn(SB²)(H₂O)₂]_n are 5.67 and 6.08 B.M., respectively.

These values are within the limits for Mn(II) species with spin-free five unpaired electrons, and indicate that the new coordination polymers are high-spin d⁵-systems, presumably with octahedral geometry (37). The magnetic moments of the two Cu(II) coordination polymers are 1.93 and 1.80 B.M., respectively, and approximate the spin-only value (1.73

Table 4. Thermogravimetric data^a

Coordination polymers	% Loss in weight found		Temp °C	Assignment of mass loss
	Found	(Calcd.)		
[Mn(SB ¹)(H ₂ O) ₂] _n	10.64	10.68	150–180	Loss of two coordinated water molecule.
	78.99	78.95	180–800	Decomposition of ligands and formation of metal oxide.
[Co(SB ¹)(H ₂ O) ₂] _n	10.51	10.55	150–180	Loss of two coordinated water molecule.
	75.63	75.68	180–800	Decomposition of ligands and formation of metal oxide.
[Ni(SB ¹)(H ₂ O) ₂] _n	10.59	10.56	150–180	Loss of two coordinated water molecule.
	77.97	78.08	180–800	Decomposition of ligands and formation of metal oxide.
[Cu(SB ¹)(H ₂ O) ₂] _n	10.47	10.41	150–180	Loss of two coordinated water molecule.
	76.92	76.98	180–800	Decomposition of ligands and formation of metal oxide.
[Zn(SB ¹)(H ₂ O) ₂] _n	10.35	10.36	150–180	Loss of two coordinated water molecule.
	76.54	76.57	180–800	Decomposition of ligands and formation of metal oxide.
[Cd(SB ¹)(H ₂ O) ₂] _n	9.17	9.12	150–180	Loss of two coordinated water molecule.
	67.48	67.45	180–800	Decomposition of ligands and formation of metal oxide.
[Mn(SB ²)(H ₂ O) ₂] _n	9.83	9.86	150–180	Loss of two coordinated water molecule.
	80.54	80.57	180–800	Decomposition of ligands and formation of metal oxide.
[Co(SB ²)(H ₂ O) ₂] _n	9.71	9.75	150–180	Loss of two coordinated water molecule.
	77.51	77.53	180–800	Decomposition of ligands and formation of metal oxide.
[Ni(SB ²)(H ₂ O) ₂] _n	9.79	9.76	150–180	Loss of two coordinated water molecule.
	79.76	79.74	180–800	Decomposition of ligands and formation of metal oxide.
[Cu(SB ²)(H ₂ O) ₂] _n	9.67	9.63	150–180	Loss of two coordinated water molecule.
	78.79	78.71	180–800	Decomposition of ligands and formation of metal oxide.
[Zn(SB ²)(H ₂ O) ₂] _n	9.53	9.58	150–180	Loss of two coordinated water molecule.
	78.37	78.32	180–800	Decomposition of ligands and formation of metal oxide.
[Cd(SB ²)(H ₂ O) ₂] _n	8.54	8.52	150–180	Loss of two coordinated water molecule.
	69.64	69.61	180–800	Decomposition of ligands and formation of metal oxide.

^aSB¹ = N,N'-di(carboxymethylene)terephthalaldehydediimine.

SB² = N,N'-di(carboxyethylene)terephthalaldehydediimine.

B.M.) expected for one unpaired electron. It is suggested that these complexes also have octahedral geometry (38). The magnetic moments of the Co(II) coordination polymers are 3.64 and 4.03 B.M. These values suggest a high-spin octahedral environment for the Co(II) (39). The magnetic moments of Ni(II) coordination polymers are 2.97 and 2.90 B.M., and are in the expected range for similar hexacoordinated Ni(II) ions (40). Finally, Zn(II) and Cd(II) coordination polymers are diamagnetic as expected for d^{10} -systems. Presumably, these species have an octahedral geometry.

3.5 Thermogravimetric Analysis

The TGA curves were obtained at a heating rate of $10^{\circ}\text{C}/\text{min}$ under N_2 in the temperature range, $50\text{--}800^{\circ}\text{C}$. The thermal degradation techniques like TG, DTA, and DSC provide much information about the coordination compounds. Indeed, in the present study, the thermal traces are very useful in further information of the number and nature of water molecules present and also helpful to propose the intermediates and final product formed during their decomposition. Thermogravimetric data are summarized in Table 4. In all materials, decomposition occurred in two steps. The first step occurs between $150\text{--}180^{\circ}\text{C}$ and shows a mass loss corresponding to two water molecules (41). It is suggested that the water molecules are coordinated to the metal ion (42). The second step, which occurs between $180\text{--}800^{\circ}\text{C}$, exhibits a loss of mass corresponding to the ligand molecules. The complexes are stable up to 450°C . The products at 800°C are the corresponding metal oxides.

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